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The optimization of the properties of the multicomponent PZT type ceramics by the choice of appropriate technological conditions

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Abstract. By the conventional ceramic sintering method, doped lead zirconate titanate (PZT) samples with the chemical composition **0.55PbTiO₃-0.43PbZrO₃-0.02[Pb(Cd_{0.5}W_{0.5})O₃]** have been obtained from oxides PbO, ZrO₂, TiO₂, CdO, WO₃. For the samples of A series, synthesis was led in the 1223 K temperature by 3 hours and in the case of the samples of B series, by 6 hours in the same temperature but in two parts (each 3 hours long). Both series were sintered in the 1423 K temperature by 6 hours. All samples were subjected to polarization ($T_{pol.}=423$ K, $E_{pol.}=30$ kV/cm, $t_{pol.}=30$ min.) and next dielectric ($\epsilon^T_{33}/\epsilon_0$, $\text{tg}\delta$) properties were measured for the samples in the shape of discs. For the samples of B series, larger values of the dielectric permittivity $\epsilon^T_{33}/\epsilon_0$ were observed, as well as lower values of the dielectric loss $\text{tg}\delta$.

1. INTRODUCTION

The PZT-type materials are commonly used in engineering. They are used as transducers between mechanical and electrical energy, such as phonographs, pickups, air transducers, underwater sound and ultrasonic generators, delay-line transducers, wave filters, piezoelectric micromotors, microrobots, actuators and etc [1, 2]. The achievements of the technology and researches of the new ceramic ferroelectric materials leads to progress in the modern piezoelectronic. The exploration and study of the piezoelectric effect in PZT ceramics make huge possibility in building piezoceramic electronic elements. The increasing possibilities of applications of these materials are connected with both the selection and improvement of the structure and microstructure (decreasing porosity, increasing density, decreasing grain dimensions) by means of choice of the suitable technological conditions [3-7].

2. EXPERIMENTAL PROCEDURES

The ceramics with following chemical composition 0,55PbTiO₃-0,43PbZrO₃-0,02[Pb(Cd_{0.5}W_{0.5})O₃] has been investigated. Two series of sample (A and B) were obtained by the conventional ceramic sintering method (CCS). The samples were obtained from oxides PbO, ZrO₂, TiO₂, CdO, WO₃ milled through 15 hours. In the case of series A synthesis were made in 1223 K temperature for 3 hours, however, series B synthesis were made in 1223 K through 6 hours (two parts, each 3 hours long). Both series were sintered in 1423 K temperature through 6 hours.

A very important question in the technology of ferroelectric materials is how to provide consistence in the stoichiometry between product obtained and chemical composition described by a molecular formula of the compounds. It's of great importance in the case of ceramic ferroelectrics containing lead [8]. Therefore sintering were made in presence of the casting powder with the same chemical composition as investigated material with 5% excess of PbO.

The samples were ground and polished. Electrodes on their surface were deposited by the silver paste burning method. All samples were subjected to polarization by the low temperature method ($T_{pol}=423$ K, $E_p=30$ kV/cm, $t_{pol}=30$ min). Ceramic samples in the shape of discs were used to measure their dielectric properties (capacity bridge of the BM 595 Tesla type). The grain size of the ceramics was observed using a scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSIONS

Measurements of dielectric permittivity ϵ and tangent of dielectric loss of angle $\text{tg}\delta$ were obtained for 55% mol PbTiO_3 ceramic materials to show the influence of the conditions of synthesis on their dielectric properties. All curves of dielectric permittivity obtained in various frequencies for ceramics with different times of synthesis A (3 hours) have clear maximum connected with phase transition which takes place at the temperature of 586 K (fig. 1).

The lack of temperature displacement of maximum of dielectric permittivity with changing frequencies proves that transition between ferroelectric and paraelectric phase is the 1st type of transformation. The same situation was observed for ceramic samples B (6 hours of synthesis).

The samples A with synthesis time 3h have lower values of the dielectric permittivity and much higher values of the tangent of dielectric loss of angle both in the room and in the Curie temperatures. It was also observe that transition takes place in wider range of temperatures and the peak of dielectric permittivity become wider. It was caused by not complete reaction of components (oxides) and it was a result of short time synthesis, too.

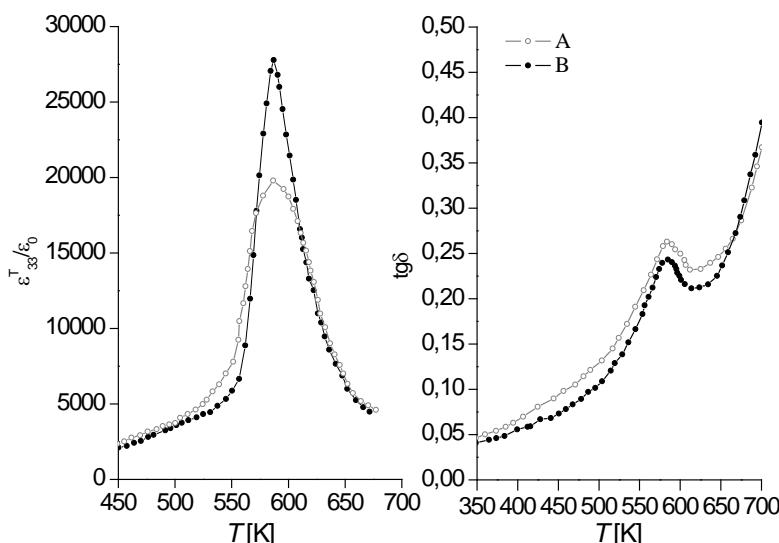


Figure 1. The temperature dependences of dielectric permittivity and tangent of dielectric loss of angle for samples with different synthesis time A (3h) and B (6h).

The structure observed by SEM shows (**Fig. 2a**) large grains (about 20 μm) for samples A. **Fig. 2b** shows a more homogeneous structure of fine grain size (about 10 μm) for samples B.

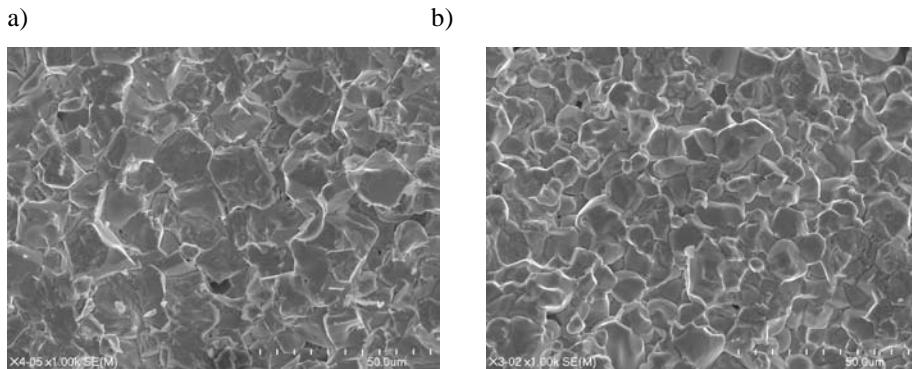


Figure 2. The SEM images from the crack of the samples: a) A (3h long synthesis), b) B (6h long synthesis).

4. CONCLUSIONS

1. An increase of the synthesis time in the PZT type ceramics has an influence on grains size and development of grains.
2. Longer synthesis time lead to obtain materials with better dielectric properties such as permittivity and tangent of dielectric loss of angle.
3. Changes in the time of synthesis in multicomponent ceramics of the PZT type lead to obtain materials for different range of technical applications.

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